

Non-destructive XRD Analysis on Micro-samples of Cultural Heritage and Archaeological Materials

Chiara Mangano¹, Benedetta Chrappan Soldavini¹, Cassandra Savoldi¹, Marta Sardara², Armida Sodo², Carlo Meneghini², Bobby Joseph³, Marco Merlini¹

¹Department of Earth Sciences, University of Milan, Milan, Italy

²Department of Sciences, University of Roma Tre, Roma, Italy

³Elettra Sincrotrone Trieste, Italy

Presenting author email: chiara.mangano@unimi.it

In the field of cultural heritage diagnostics and archaeometry, there is a growing demand for analytical techniques capable of operating in a fully non-invasive in-situ mode or, more frequently, in a minimally invasive and non-destructive fashion. Micro-sampling - often authorized by heritage authorities - is commonly employed to enable accurate material characterization using advanced analytical instrumentation, offering significant advantages in data quality over portable devices.

Among such techniques, X-ray diffraction (XRD) stands out, alongside spectroscopy methods such as XRF, EPMA, Raman, and IR. This contribution presents phase characterization results obtained by X-ray powder diffraction (XRPD) in three case studies: (1) analysis of pigments in painted decorations, (2) altered metallic components from pipe organs, and (3) archaeological ceramics.

Non-destructive XRD analyses were performed on micro-samples: $\sim 50 \times 50 \times 50 \mu\text{m}^3$ fragments of paint layers collected non-invasively; sub-millimetric metal fragments with surface corrosion; and ~ 10 mg of ceramic material from archaeological finds and earthy incrustation. Data were collected both in the laboratory—using Mo and Cu microfocus sources (Rigaku Synergy instrument with area detector)—and at the synchrotron radiation facility (XPRESS beamline, Elettra), under high-resolution conditions ($\lambda = 0.4975 \text{ \AA}$, Pilatus 6M detector, sample-to-detector distance 940 mm).

Synchrotron-based analyses enabled the identification of crystalline phases present in quantities as low as 0.1 wt%, especially in metal corrosion layers and pigments, highlighting phases not clearly detectable in laboratory spectra. In parallel, Synchrotron reference data proved essential for interpreting minor peaks observed in lab datasets, confirming the relevance of synchrotron calibration in such studies. Laboratory microfocus sources—especially Cu-based—also showed promising performance.

This comparative study supports the development of a robust analytical protocol, where synchrotron-based diffraction plays a critical role in guiding the interpretation of large-scale laboratory analyses, enabling broader applications in cultural heritage diagnostics without compromising material integrity or logistical feasibility.